2. EXPERIMENTAL TESTS

Six types of experimental tests were conducted to determine the reactivity of nitrate salts with oil, charcoal, graphite, and rags when heated at the rate expected during ISV processing. The test types, their purpose, and the test numbers of the individual tests that were performed are listed below. Although the test numbers were assigned sequentially by the EMRTC, the tests for this study are not ordered sequentially but by the scale up of the volume of the experimental ingredients. The tests in this section are discussed in this scale-up order. The sequential test numbers were retained to enhance communication with the subcontractor performing the tests, EMRTC of the NMT.

- **Henken Tests:** Determine (in general terms) the temperature at which the mixture of nitrate salts with powdered cellulose becomes explosive (see Section 2.1)

  Tests 1, 2, 3, 4, 5, and 6

- **Koenen Tests:** Determine (in general terms) whether mixtures of nitrate salts with charcoal, graphite, and powdered cellulose react violently under confinement (see Section 2.2)

  Tests 1, 2, 3, 4, and 5

- **Burn-Rate Tests:** Demonstrate propagation with mixtures of nitrates with charcoal derived from pyrolyzed rags and with nitrate-soaked rags (see Section 2.3)

  Tests 12a, 12b, 12c, 12d, 12e, and 12f

- **Five-gal Cook-Off Tests:** Determine whether reactions between nitrates and the above mentioned materials occur under extreme thermal conditions (see Section 2.4)

  Tests 1, 2, 3, 4, 5, 6, 7, 8, 10, 11, 14, 15, 16, 18, 19, 20, and 22

- **Fifty-Five-gal Cook-Off Tests:** Determine the possibility of runaway reactions of the materials when heated to an elevated temperature (see Section 2.5)

  Tests 9, 13, 17, 21, and 23

- **Fifty-Five-gal Drum Containment and Mitigation Tests:** Determine, under realistic conditions, the effectiveness of mitigative measures (see Section 2.6)

  Tests 24, 25, 26, and 27.

The experimental details provided in this section were excerpted, with extensive editing, from the NMT test reports (EMRTC 2000, 2001). The test mixtures were comprised of the following nitrate and fuel materials:

Base mixture. The base mixture (i.e., base mix) was formulated to represent the nitrate salt cake in the buried waste, and consisted of 60% sodium nitrate ($NaNO_3$), 30% potassium nitrate (KNO_3), 3% sodium chloride (NaCl), 3% sodium sulfate (Na_2SO_4), 1% monobasic sodium phosphate (Na_2HPO_4), 1% sodium bicarbonate ($NaHCO_3$), 0.5% sodium fluoride (NaF), 0.5% sodium nitrite ($NaNO_2$) and 1% water (H_2O). The trace constituents were added to allow postulated effects, such as catalytic oxidation by nitrite, and bubble formation by bicarbonate decomposition. The individual chemicals were ground to a particle size of 100 to 250 mesh (150 to 63 μ) using industrial sized burr coffee grinders. The

material was placed in 5-gal totes normally used to mix cement in small batches. The totes were placed on motor-driven rollers for mixing.

Oil. The oil used was Texaco Regal R&O 32 machine oil, purchased from a local supplier in 5-gal buckets.

Cellulose. Cellulose was used to describe the combination of 50% cotton rags and 50% Kimwipes. The cotton rags were washed and sorted by the supplier. Most were used (i.e., worn) towels. The Kimwipes were purchased from a chemical supply house.

Powdered cellulose. Powdered cellulose was used in the Phase I Henkin, Koenen, and first 5-gal cook-off tests. The cellulose material was a fine-ground commercial material furnished by the Aldrich Chemical Company.

Nitrate solution. This is a saturated aqueous solution of base mix at 60°C. Cellulose was soaked in this solution to make the nitrate-treated material for the tests.

Treated cellulose procedure. A simple procedure was used to treat the cellulose. After establishing the proper mass of both base mix and cellulose needed for one batch, the base mix was mixed with just enough water to saturate the cellulose. The aqueous solution was heated to 60°C to dissolve the salts and form a saturated aqueous solution. The solution was evenly soaked into the cellulose. The mixture was then allowed to cool. Using this process, the cellulose would absorb all of the solution, thus eliminating the need to weigh and recalculate the nitrate concentration of the batch. If dry material was desired, the treated cellulose was placed on screens and allowed to air dry.

Charcoal. The 80-mesh (180-μ) powdered charcoal was purchased from Skylighter, Inc.

Carbonized cellulose. The cellulose was carbonized by placing it in a closed 5-gal drum and heating at 400°C for 6 hours. The batch was then allowed to cool overnight before the drum was opened.

2.1 Henkin Tests

Using the Henkin-McGill (1952) critical temperature test, Tc (i.e., the lowest temperature at which an energetic material exhibits thermal runaway) and the time-to-explosion at various temperatures were determined using an apparatus (see Figure A-1 of Appendix A) similar to those described by Rogers (1975), Olson and Block-Boldten (1995), and Olson and Banks (1994).

Samples of each test material were sealed inside aluminum blasting-cap shells using a hand-operated hydraulic press and specially made hollow aluminum plugs to form a seal with the shell. This gave the nominal 46-mg samples the geometry of a thin disc with a diameter of 0.65 cm (0.25 in.) and a thickness of 0.7 cm (0.28 in.). The exact thickness of each sample was measured.

An Omega Engineering, Model 920 proportional controller controlled the temperature of the Wood's metal bath[a]. The actual bath temperature was measured immediately before each test using an Omega K-type stainless steel sheathed thermocouple and a Fluke Model 52 digital readout. The sample

a. A Wood's metal bath is used in place of an oil bath when temperatures higher the 250°C are required. The alloy comprises 50% bismuth, 25% lead, 12.5% tin, and 12.5% cadmium.

was held in a heavy lid and lowered into the hot bath by remote operation using an air-driven piston and cylinder. The time-to-explosion was measured using a digital stopwatch from the initial time the sample was immersed in the molten metal bath to the time, if any, at which the sample holder ruptured. The timing uncertainty was estimated to be about 1 second.

To find the critical temperature, the time-to-explosion tests were repeated at various bath temperatures until the minimum temperature for thermal runaway and shell rupture was identified. The Wood's metal bath has a maximum temperature limit of 400°C.

The following samples were prepared for the Henkin tests:

- **Test 1** Base mix (91%) with Regal R&O 32 (9%)
- **Test 2** Base mix (90%) with Regal R&O 32 (10%)
- **Test 3** Base mix (87%) with Regal R&O 32 (13%)
- **Test 4** Base mix (80%) with powdered charcoal (20%)
- **Test 5** Base mix (80%) with graphite (20%)
- **Test 6** Base mix (65%) with powdered cellulose material (35%), pre-dried.

The material was hand mixed by a Torres Laboratory technician.

2.1.1 Results

Henkin tests were performed on the six mixtures listed in Section 2.1. The tests were run at temperatures ranging from 185 to 400°C, with the Test 6 mixture being the only mixture that responded to the test. The critical temperature of the Test 6 mixture was determined to be 339°C using the Bruceton up/down method (Henkin-McGill 1952). It is evident that the critical temperature for the rest of the mixtures was higher than 400°C for the diameter used in the Henkin test. Table 1 lists the results of the individual tests.

Table 1. Henkin test results.

Test Series	Base mix with the following:	Critical Temperature (°C)	Time to Explosion (seconds)	Apparent Density (g/cm^3)
1	9% oil	>398	>600	172
2	10% oil	>399	>600	2.07
3	13% oil	>398	>600	1.67
4	20% powdered charcoal	>398	>600	1.89
5	20% graphite	>398	>600	1.81
6	20% powdered cellulose	339	190	1.85

2.2 Koenen Tests

The apparatus consists of a nonreusable steel tube (75 mm [3 in.] long with a 24-mm [0.9-in.] inside diameter [ID] and 0.60-mm [0.024-in.] wall thickness), with a reusable closing device, installed in a heating and protecting fixture. The Koenen test is one of many used to determine the UN classification of a sample material. In this test series, it was chosen to determine if the material produces violent effects when heated under confinement. A full explanation of the Koenen test and procedures can be found in the *UN Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria* (United Nations 1999).

To determine whether a cook-off temperature exists under Koenen test conditions, one test on each sample material was performed with a thermocouple placed in the center of the sample on the axis. The orifice was machined with a 1.59-mm (0.0625-in.) national pipe-threaded hole in the center and 6.35-mm (0.25-in.) holes drilled in what remained of each of the four quadrants. Data were collected at 200 Hz using *Workbench for Windows* Strawberry Tree commercial software (IOtech/Strawberry Tree 1996) and an IOtech Dynares input/output (I/O) board mounted in a Pentium computer.

The Koenen test proved to be an unreliable method for determining the cook-off temperature of sample material approximately 25 mm (1 in.) in diameter because the heating was too fast to measure runaway conditions. However, it did prove valuable as a method for predicting the orifice diameter at which the material would rupture the Koenen tube. From the reaction profile collected during the 20-mm (0.787-in.) orifice test run, a rate of reaction could be calculated. The rate of reaction was then correlated against orifice diameter to predict the diameter at which the tube would rupture. Using this method, the number of individual tests was reduced to five.

The Koenen test mixtures were prepared as follows:

- **Test 1** Base mix (90%) with Regal R&O 32 (10%)
- **Test 2** Base mix (87%) with Regal R&O 32 (6.5%) and CCl_4 (6.5%)
- **Test 3** Base mix (80%) with powdered charcoal (20%)
- **Test 4** Base mix (80%) with graphite (20%)
- **Test 5** Base mix (65%) with powdered cellulose material (35%), pre-dried.

2.2.1 Results

The results of these tests can be seen in Table 2. The limiting diameter and the burn-rate are shown. The limiting diameter is used to judge the sensitivity of an energetic material to intense heat under confinement. If the limiting diameter (i.e., orifice diameter) was less than 1 mm (0.04 in.), the material would not be judged energetic for this test.

The test procedure dictates that the burn lasts for 5 minutes, although the test may be terminated after the material has reacted. With the use of a thermocouple placed in the center of the sample, the time necessary to consume all of the material could be easily determined and recording the temperature data (at 20 mm [0.787 in.]) for the full 5 minutes would not be necessary.

Table 2. Results from the Koenen test series.

Test Series	Base Mix[a] with the following:	Orifice Diameter (mm)	Effects Ref.	Limiting Diameter (mm)	Peak Temp Measured (°C)	Rate of Reaction (°C/sec)	Sample Remaining
1	10% oil	20	O				11.5
		12	O				15.7
		8	O				32.0
		5	O				23.0
		1	O	<1	NA	7.2	11.8
2	6.5% oil and 6.5% CCl_4	20	O				8.5
		2	O				26.4
		1	O	<1	NA	7.1	23.8
3	20% powdered charcoal	20	O				
		2	F				
		5	B				
		3	F	3	1,185	169.7	N/A
		5	O				
		5	O				
4	20% graphite	20	O				
		5	O				
		1.5	F				
		3	F	3	1,205	144.0	N/A
		5	O				
		5	O				
5	35% powdered cellulose	20	O				
		5	O				
		3	F	3	1,049	155.0	N/A
		5	A				
		5	B				

a. This consisted of 60% sodium nitrate ($NaNO_3$), 30% potassium nitrate (KNO_3), 3% sodium chloride (NaCl), 3% sodium sulfate (Na_2SO_4), 1% monobasic sodium phosphate (Na_2HPO_4), 1% sodium bicarbonate ($NaHCO_3$), 0.5% sodium fluoride (NaF), 0.5% sodium nitrate ($NaNO_2$), and 1% water (H_2O).

Effects:

"O": Tube unchanged;

"A": Bottom of tube bulged out;

"B": Bottom and wall of tube bulged out;

"F": Tube fragmented into three or more mainly large pieces, which in some cases may be connected with each other by a narrow strip.

N/A: Not Applicable

Temperature data were only recorded during the first test of each series. The 20-mm (0.787-in.) orifice with the thermocouple was used for this test. The results of the temperature data are shown in Figures B-1 and B-2 of Appendix B. Although the profiles were intended to show the temperature at the point of reaction, the most that can be obtained from the data are the burn-rate and the peak temperature. Both profiles did identify a critical temperature at this diameter and may be useful for other purposes.

The results of the base mix and oil test (Test 1) were similar to the base mix, oil, and CCl_4 test (Test 2). Both tests resulted in a limiting diameter of less than 1 mm (0.04 in.). Neither of these tests

fractured the Koenen tubes. For Test 2, the R&O oil content was reduced to 6.5%, and 6.5% CCl_4 was added.

The temperature profiles shown in Figure B-1 of Appendix B can be misleading. The profile leads us to believe that the reaction started at 66°C, ended at about 245°C, and had an intermediate reaction at 175°C. Because of the intense heat being transferred into the sample by the four propane burners, the outside of the sample would be much hotter than the temperature recorded at the center. Although it may be possible to find the critical temperature from the results of the Koenen test, it would first be necessary to perform cook-off experiments for calibration purposes. The temperature profile was of interest in determining how the rate of reaction relates to the limiting diameter.

Tests 3, 4, and 5 resulted in a limiting diameter of 3 mm (0.118 in.), meaning the reaction fragmented the Koenen tubes. At an orifice size of 5 mm (0.196 in.), the tubes were not fragmented, but the bottom or sides may have bulged. If the tests were run for UN qualification of the sample material, the results would have been positive, meaning the material showed a violent effect on heating under confinement. The results can be found in Table 2. The particle size of the material could enter into the results; however, no tests were performed to determine the effect of particle size on the limiting diameter of the samples.

The temperature profiles for tests 3, 4, and 5 are found in Figure B-2 of Appendix B. There was no way to identify a critical temperature from the profiles. The rate of reaction was fast, with a peak temperature in the 1,200°C range. It was significant that all three materials shattered the tube at a 3-mm (0.12 in.) orifice because all three failed the UN test for explosive sensitivity.

It may be noticed that Test 4 was started at an above-ambient temperature (57°C). The preheating was caused by the pilot light being set too high.

2.3 Burn-Rate Tests

The burn-rate tests were performed in 914-mm (36-in.) long, schedule-40 black pipe with a 51-mm (2-in.) ID. Thermocouples were placed every 102 mm (4 in.). Seven thermocouples were spaced over the first 762 mm (30 in.) at 102-mm (4-in.) intervals, leaving the upper 152 mm (6 in.) for the igniter. An eighth thermocouple was used to record the axial temperature near the center of the pipe. The same computer system used in the Koenen tests collected the temperature data from the thermocouples.

The tests were ignited at an elevated temperature. To heat the test, one silicone rubber tape heater was spiral-wound around the pipe. To speed the heating rate, two layers of 8-lb insulation were used to cover the heating tape. One stick-on thermocouple was placed between the heating tape and the pipe wall for temperature control.

The tests were performed in the vertical position with the igniter on the top. The burn-rate fixtures were taped to a (nominal) 2 × 4-in. board. The bottom of the pipe was closed using a piece of Kimwipes, and a hot-wire igniter was placed in the top. The igniter was made from resistance wire and bench tested, using a Variac's power source, to maintain the resistance wire at a "white hot" temperature. The igniter was made with five loops of the resistance wire wound around a 13-mm (0.5-in.) drill bit shank. The Variac power source, set to 17 V, furnished AC power to the igniter.

2.3.1 Results

Six burn-rate tests were performed (see Table 3). With the exception of Test 12a base mix (90%) and oil (10%), the ignition of all the series could be obtained with a hot wire alone. Nitrate-treated rags are very adaptable as a fuse and replaced the (fast burning) granulation (FFFg) gunpowder in the following tests. Figures B-14 through B-18 of Appendix B show the temperature profiles of tests12b through 12f. The tests were performed at elevated temperatures (a more sensitive condition than ambient), however, it was necessary to test below the ignition temperature of FFFg gunpowder.

2.3.1.1 Test 12a. The first test with base mix and oil would not ignite with a hot wire alone. The addition of FFFg gunpowder did not provide ignition, but did compact the material in the pipe. A mixture of treated cellulose, carbonized cellulose, and a few grams of the FFFg powder also failed to ignite the sample. A new pipe was loaded with the same mixture and various methods of ignition were tried with no success. It was not possible to ignite and burn a mixture of base mix and oil using common ignition methods. Ignition was achieved in one test, but the mixture channeled and did not propagate.

2.3.1.2 Test 12b. Carbonized cellulose (20%) mixed manually with base mix (80%) was heated to an internal temperature of 170°C and ignited. The average burn-rate was determined to be 135 mm (5.33 in.) per second.

2.3.1.3 Test 12c. A mixture of cellulose treated with a saturated aqueous solution of base mix, to give a nominal 65-wt% nitrate when dried, was heated to an internal temperature of 148°C and ignited. The average burn-rate was determined to be 156 mm (6.15 in.) per second.

2.3.1.4 Test 12d. A mixture of cellulose treated with a saturated aqueous solution of base mix, to give a nominal 55-wt% nitrate when dried, was heated to an internal temperature of 186°C and ignited. The average burn-rate was determined to be 156 mm (6.15 in.) per second.

2.3.1.5 Test 12e. Carbonized cellulose (32%) mixed manually with base mix (68%) was heated to an internal temperature of 202°C and ignited. The average burn-rate was determined to be 8.4 mm (0.33 in.) per second.

2.3.1.6 Test 12f. This test was set up slightly different than the above tests. The burn-rate tube was filled to 75% capacity of a mixture of cellulose treated with a saturated aqueous solution of base mix, to give a nominal 65-wt% nitrate when dried, was then wetted and mixed with water from a spray bottle to obtain a nominal moisture content of 10%. The top 25% of the burn-rate tube was then filled with the dry, treated cellulose mixture, and this top 25% was then heated to 140°C and ignited. The burn-rate for the wetted mixture was then determined. Surprisingly, the average burn was determined to be 26 mm (1.03 in.) per second. This was much faster than the same dry mixture. See Table 3 for a complete summary of these test results.

Table 3. Results from the burn-rate tests, Tests 12a to 12f.

Test	Base Mix[a] with the following:	Chemical	Time of Arrival (seconds)	Δx in.	Δt s	Velocity (in./second)
Test 12a	10% oil		No Burn			
Test 12b	20% carbonized rags and Kimwipes	1	2,000.00			
		5	2,003.00	16	3.00	5.33
		6	2,015.00	4	12.00	0.33
		7	2,024.00	4	9.00	0.44
Test 12c	35% impregnated rags and Kimwipes	3	646.40			
		4	647.20	4	0.80	5.00
		6	648.50	8	1.30	6.15
		7	650.10	4	1.60	2.50
Test 12d	45% impregnated rags and Kimwipes	1	10.94			
		2	11.53	4	0.58	6.86
		4	12.43	8	0.91	8.84
		6	13.62	8	1.19	6.72
		7	16.41	4	1.79	2.23
Test 12e	32% carbonized rags and Kimwipes	1	33.00			
		2	67.91	4	34.90	0.11
		3	88.31	4	20.40	0.20
		4	116.03	4	27.72	0.14
		5	153.63	4	37.60	0.11
		6	165.82	4	12.19	0.33
		7	184.68	4	18.86	0.21
Test 12f	35% impregnated rags and Kimwipes (10% water was added to mix)	1	43.63			
		2	47.03	4	3.40	1.18
		4	53.64	8	6.61	1.21
		7	70.70	12	17.06	0.70

a. This consisted of 60% sodium nitrate ($NaNO_3$), 30% potassium nitrate (KNO_3), 3% sodium chloride (NaCl), 3% sodium sulfate (Na_2SO_4), 1% monobasic sodium phosphate (Na_2HPO_4), 1% sodium bicarbonate ($NaHCO_3$), 0.5% sodium fluoride (NaF), 0.5% sodium nitrate ($NaNO_2$), and 1% water (H_2O).

2.4 Five-gal Cook-Off Tests

Two types of 5-gal drums were used for these tests. The cold-steel drums were 356-mm (14-in.) ID and the stainless steel drums were 267-mm (10.5-in.) ID. The stainless steel drums were furnished by the INEEL and the cold-steel drums were purchased from a commercial barrel supplier. Both were stamped as approved Department of Transportation (DOT) shipping containers.

Two types of band heaters were used for the tests. The first five tests were run using Cole-Parmer Dual-Element Heating Tapes (PN P36050-85) with double insulation. The remaining tests used either a Chromalox Model DBW (2,900 W) or Model HB (1,200 W) two-piece band heaters, purchased from E&M Sales or Omega Engineering. Either two DBW or three HB heaters were used for each test. Four of the 1,200-W tape heaters were used on each drum.

Type-K thermocouples were purchased from Omega Engineering and sent to the INEEL for calibration verification. One thermocouple was placed between the bottom heater and the outside wall of the drum for the controller. Three thermocouples were placed through the lid of the container reaching about 67% of the way into the sample material. The interior thermocouples were placed in the center (axial), mid-radius, and radius (close to the inside wall).

A vent pipe (38 mm [1.5 in.] ID) was installed into the lid of each test. Excluding the first four tests, one thermocouple was located in the vent pipe to sample the temperature of the vent gases. Using a system of elbows and nipples, any vapor or liquid material ejected from the experiment would be contained in a waste bucket. The waste bucket was designed to protect the environment and to allow for collecting, sampling, and measuring of sample materials vented during the test. During the first four tests, a piece of PVC pipe was used with unsuccessful results.

Each test was wrapped with three layers of 8-lb, high-density fiberglass insulation and two layers of foil-backed glass insulation of the same density. Glass tape was used to keep the insulation in place until the final layer was wrapped and taped with duct tape.

The temperature data were collected using a Pentium computer containing an IOtech Dynares I/O interface board connected to a TC terminal panel. This computer was also used to control the rate of heating with Strawberry Tree commercial software (IOtech/Strawberry Tree 1996). The sampling rate was at 1-second intervals. The software was set to reproduce the ISV heating rate of 100°C per hour between ambient and 500°C, then to maintain 500°C until the tests were terminated. The actual heating rate experienced by the drum contents lagged the set rate, varied with the materials and the position in the drum, and often did not achieve the desired 100°C per hour.

An accelerometer was attached to a 762-mm (30-in.) steel tripod and placed on the lid of each drum. Four Model-3010 accelerometers were purchased from PCB Industries and were calibrated before leaving the factory. The accelerometer in each test was connected to a second Pentium computer containing a similar Dynares interface board. The data were collected using *Workbench for Windows* software (IOtech/Strawberry Tree 1996) at 100 Hz. A faster rate would have been better, however, the files quickly became too large to be reasonably handled.

Tests 1 through 4 were run using the insulated heat tapes. Because the results of these tests were less than ideal, Test 5 (sand and oil) was run to check out the system. These tests were buried under 610 mm (2 ft) of sand. Tests 6 through 8 were run using the band heaters. This type of heater handled the pressure from being buried better than the tape heaters. The band heaters were rated to 450°C, but were heated to 700°C under runaway conditions with no apparent damage. The tape heaters were rated to 720°C.

Air sampling was attempted on the first eight tests. A Draeger Safety Model 23 Multiwarn II was used to perform the air analysis for carbon monoxide (CO), carbon dioxide (CO_2), nitric oxide (NO), and nitrogen dioxide (NO_2). This sampling unit has a built-in pump and a digital readout and downloads the collected data to a PC. The gas analysis was not successful because of the high concentration of the gases pumped from the drums. The headspace of the drums would fill with a very concentrated gas and when the concentrated gas was pulled into the analyzer, it would immediately saturate the sensors. Even at a dilution of 100:1 (using bottled air), it was not possible to sample for any extended period of time. The purpose was to measure the concentration of the gases at defined periods of time during the test. The time period during the 100°C per hour heating ramp was of particular interest, as that was the period simulating the 100°C per hour heating rate of ISV. The generation of gases was an indication of a chemical reaction.

Unfortunately, after a few minutes of sampling, the unit was saturated and no data could be collected. Proper gas collection and analysis would be better suited to a small-scale test with no headspace to trap the gases.

Four 5-gal tests were planned for this series (see Figures A-14 through A-19 of Appendix A), but additional tests were performed as a method to test suitability of the equipment for this type of testing. The base mix was prepared (as described above) and used in the following combinations:

- **Test 1** Base mix (90%) with Regal R&O 32 (10%)
- **Test 2** Base mix (87%) with Regal R&O 32 (6.5%) and CCl_4 (6.5%)
- **Test 3** Base mix (80%) with powdered charcoal (20%)
- **Test 4** Base mix (65%) with powdered cellulose material (35%), pre-dried
- **Test 5** Sand (90%) with Regal R&O 32 (10%)
- **Test 6** Sand (90%) with Regal R&O 32 (10%)
- **Test 7** Base mix (90%) with Regal R&O 32 (10%)
- **Test 8** Base mix (90%) with Regal R&O 32 (5%) and CCl_4 (5%)
- **Test 10** A saturated, aqueous solution of the nitrate-base mixture was added to a 50% (by weight mixture) quantity of rags and Kimwipes sufficient to give a nominal 65 to 35-wt% nitrate and cellulose mixture when the water was evaporated
- **Test 11** A quantity of the nitrate-base mixture, sufficient to give a nominal 65 to 35-wt% nitrate and cellulose ratio, was placed on top of rags and Kimwipes compressed in a 5-gal stainless steel drum
- **Test 14** A quantity of the nitrate-base mixture, sufficient to give a nominal 80 to 20-wt% nitrate and carbon ratio, was placed on top of unground, carbonized-cellulose material in a 5-gal stainless steel drum. The carbonized material was prepared by the pyrolysis of a 50% mixture of rags and Kimwipes
- **Test 15** Base mix with no fuel was used for this test. A cold-steel drum was tested to determine the effects of the molten nitrate mixture alone on the container
- **Test 16** A quantity of the nitrate-base mixture, sufficient to give a nominal 80 to 20-wt% nitrate and carbon ratio, was placed on top of 80-mesh graphite in a 5-gal stainless steel drum
- **Test 18** A saturated, aqueous solution of the nitrate-base mixture was added to a 50% (by weight mixture) quantity of rags and Kimwipes sufficient to give a nominal 37.5 to 62.5-wt% nitrate and cellulose mixture when the water was evaporated
- **Test 19** A quantity of the nitrate-base mixture, sufficient to give a nominal 80 to 20-wt% base mix and carbon ratio, was mixed with 80-mesh graphite by 100 turns in the mixing drum

- **Test 20** A quantity of the nitrate-base mixture, sufficient to give a nominal 60 to 40-wt% base mix and cellulose ratio, was placed on top of rags and Kimwipes compressed in a 5-gal stainless steel drum

- **Test 22** A 50 to 50-wt% rag and Kimwipes mixture was treated with a saturated, aqueous solution of nitrate-base mix to give a nominal 35% rags and 65% base mix ratio after the rags were dried.

The purpose of these tests was to provide detailed information on any reaction that may occur under extreme thermal conditions. These tests consisted of 5-gal drums 75% full of sample material and equipped with band heaters, controller thermocouples, internal thermocouples, and an accelerometer. Tests 15, 18, 19, and 20 were not confined. Tests 10, 11, 14, and 16 were confined in a hole full of sand with 620 mm (2 ft) of sand covering the top of the drum to simulate actual SDA conditions. In these tests, an ISV heating rate of 100°C per hour was the target heat ramp, however, the mass of the material in the drums would not heat as fast as targeted. Video footage was taken of each test.

Test 22 was set up similarly to the rest of the tests in this phase except that it was buried to the level of the drum rim only. It was heated to 150°C and the reaction was initiated with a resistance hot wire. Two video cameras were used on this test, placed approximately 15.24 and 91.44 m (50 and 300 ft) away. A 2 × 4-in. post, painted white with 30-cm (1-ft) graduations, was mounted on a stand to allow an estimate of how high the lid raised from the explosion. The top of the 2 × 4-in. post was at 3.2 m (10.5 ft).

Pictures of the tests are found in Appendix A. Table 4 lists the results of the tests (excluding the Henkin and Koenen tests).

2.4.1 Results

Tests 1 through 4 were performed simultaneously. The use of simultaneous testing was meant to save time and use the equipment to the fullest. However, the heating belts (although double-insulated, using woven fiberglass insulation recommended for use on conductive surfaces) shorted (i.e., arced) to the buckets. The pressure caused by being buried under 610 mm (2 ft) of sand probably caused the shorting. When the shorting occurred, stray electrical current found its way back to the computers and caused the data-collection boards to fail, channel by channel. The heating belts were later used on a steel drum for the carbonization process with no problems. Pictures of the tests can be found in Appendix A (see Figures A-20 through A-32).

2.4.1.1 Test 1. The Test-1 (90% nitrates and 10% oil) temperature profile contained nontypical traces indicating that the data collection was not working properly. The data were of such poor quality that they were not graphed. Although this test did not cook-off, there were holes in the 5-gal drum indicating that the heaters had arced to the steel.

2.4.1.2 Test 2. The Test-2 (87% nitrate, 6.5 % oil, and 6.5% CCl_4) temperature profile (see Figure B-3 of Appendix B) indicated that the test was heating normally until about 150 minutes. At this time, the traces started wandering and the testing ended. An inspection of the drum indicated that the heaters had shorted and arced, creating holes in the wall of the drum. The digital I/O board was ruined.

2.4.1.3 Test 3. Test 3 (80% base mix and 20% charcoal) resulted in a more normal temperature profile. There is some indication that the data collection system was being subjected to interference at about 175 minutes. The heaters did not short to the drum, so the electrical noise had to be coming from either Test 1 or 2. The temperature profile can be seen in Figure B-4 of Appendix B.

Table 4. Test summary (excluding Henkin and Koenen tests).

Test	Size	Type Drum	Base Mix (%)	Base Mix (g)	Nitrate (Actual) (%)	Form	Fuel	Fuel (%)	Fuel (g)	Fuel (Actual) (%)	Control Temp. (°C)	Peak Temp. (°C)	Time to Reaction (min.)	Results
1	5-gal	Cold steel	90	27,000	89%	Solid	Oil	10	3,000	11%	Belt heaters (arced)	---	---	No reaction
2	5-gal	Cold steel	88	27,000	87%	Solid	Oil/CCl_4	12	3,510	13%	Belt heaters (arced)	---	---	No reaction
3	5-gal	Cold steel	80	13,182	78%	Solid	Charcoal, mixed 100 turns	20	3,323	22%	338	744	214	Explosion, 63 "g"
4	5-gal	Cold steel	65	7,659	62%	Solid	Cellulose, powdered	35	4,157	38%	32	618	2.2	Explosion
5	5-gal	Cold steel	---	---	---	Solid	Sand/10% oil	---	---	---	500	---	---	Belt heaters - arced
6	5-gal	Cold steel	---	---	---	Solid	Sand/10% oil	---	---	---	500	---	---	Band heaters - OK
7	5-gal	Cold steel	90	27,000	89%	Solid	Oil	10	3,000	11%	445	-	358	Fire; bucket oxidized
8	5-gal	Stainless	90	27,000	89%	Solid	Oil (6.5%)/CCl_4 (6.5%)	10	3,000	11%	500	688	---	Smoke
9	55-gal	Cold steel	---	---	---	---	Air	---	---	---	---	---	---	Pressure release 38 psig.
10	5-gal	Stainless	65	3,117	63%	Solution	Rags/Kimwipes	35	1,678	37%	500	530	460	Fire, bulged lid and bottom.
11	5-gal	Stainless	65	3,134	63%	Solid	Rags/Kimwipes	35	1,687	37%	350	1,100	182	Bulged lid and bottom
12a	Burn pipe	2"Sch 40	90	1,595	89%	Solid	Oil	10	177	11%	---	---	---	Fizzle
12b	Burn pipe	2"Sch 40	80	456	78%	Solid	Carbonized rags/Kimwipes	20	114	22%	170	1,150	---	5.33 in./second
12c	Burn pipe	2"Sch 40	65	178	63%	Solution	Rags/Kimwipes	35	96	37%	148	948	---	6.15 in./second
12d	Burn pipe	2"Sch 40	55	164	52%	Solution	Rags/Kimwipes	45	134	48%	186	991	---	8.84 in./second
12e	Burn pipe	2"Sch 40	68	324	66%	Solid	Carbonized rags/Kimwipes	32	153	34%	202	1,155	---	0.33 in./second
12f	Burn pipe	2"Sch 40	65	178	63%	Solution	Rags/Kimwipes	35	96	37%	140	1,058	---	1.03 in/second
13	55-gal	Stainless	90	297,000	89%	Solid	Oil	10	33,000	11%	500	437	---	No reaction
14	5-gal	Stainless	81	6,657	79%	Solid	Carbonized rags/Kimwipes	19	1,554	21%	373	1,165	230	Flap in side of drum
15	5-gal	Cold steel	100	2,700	100%	Solid	None	-	-	0%	500	---	---	No reaction
16	5-gal	Stainless	80	9,126	78%	Solid	Graphite	20	2,282	22%	421	1,123	236	Drum lid melted
17	55-gal	Stainless	65	33,255	63%	Solution	Rags/Kimwipes	35	17,907	37%	206	1,200	124	Explosion, blew lid, 74 "g"
18	5-gal	Stainless	60	2,055	57%	Solution	Rags/Kimwipes	40	1,370	43%	316	1,100	168	Fireball, blew lid
19	5-gal	Stainless	80	13,587	78%	Solid	Graphite, mixed 100 turns	20	3,397	22%	500	904	393	Rapid burn and explosion
20	5-gal	Stainless	60	2,701	57%	Solid	Rags/Kimwipes	40	1,800	43%	323	1,021	174	Burn
21	55-gal	Stainless	88	101,990	87%	Solid	Carbonized rags/Kimwipes	12	13,908	13%	366	741	198	Explosion, blew lid, 79 gs
22	5-gal	Stainless	65	1,931	63%	Solution	Rags/Kimwipes	35	1,040	37%	150	---	1365	Explosion, hot wire gin.
23	55-gal	Stainless	80	68,184	78%	Solid	Carbonized rags/Kimwipes	20	17,046	22%	500	---	387	Explosion, 27 gs, 2.4 psig
24	55-gal	Cold steel	86	58,314	85%	Solid	Carbonized rags/Kimwipes	14	9,271	15%	330	---	174	Explosion, 103 "g"
25	55-gal	Cold steel	67	20,631	65%	Solution	Rags/Kimwipes	33	10,035	35%	244	---	125	Explosion, 115 gs, 68 psig
26	55-gal	Cold steel	86	175,951	85%	Solid	Graphite	14	27,945	15%	500	---	3,054	21 hr hold 200°C; Drum melted
27	55-gal	Cold steel	86	59,723	85%	Solid	Carbonized rags/Kimwipes	14	9,485	15%	314	1,100	1,001	16 hr hold 250°C; Explosion

This mixture exploded at 214 minutes and at a wall temperature at 338°C. The accelerometer registered 63 "g"[b] of acceleration (see Figure B-5 of Appendix B). The explosion blew the lid from the drum, bulged the lid outward, and bulged the bottom of the drum. Because the test was buried in sand, the drum filled with sand when the lid was blown upward. The mixture of hot nitrates, sand, and charcoal resulted in some solid formations in the bottom of the drum.

2.4.1.4 Test 4. Test 4 (a mixture of 65% base mix and 35% powdered cellulose) exploded only 2.2 minutes into the heating. Unfortunately, no reaction was expected for several hours and the video was not on to record the event. The reaction happened with the wall temperature at 32°C, which is well below all points of sensitivity of the sample material. The accelerometer registered 28 "g" of acceleration. The temperature profile indicated that the material ignited and reacted after the explosion (see Figures B-6 and B-7 of Appendix B). Also, the explosion blew the lid off the drum, bulged the lid outward, and bulged the bottom of the drum.

When inspecting the setup after it was dug up, it was apparent by the holes burned in the drum that the heaters had shorted to the drum wall. The arcing of the electrical current was the probable point of ignition that caused the reaction. The arcing burned several holes in the drum wall. The holes varied from pinholes to large holes measuring approximately 19 mm (0.75 in.)

2.4.1.5 Test 5. For Test 5, the drum was filled with sand and buried under 610 mm (2 ft) of sand. Then using the ISV ramp, the drum was cooked at 500°C. This test was designed to prove the reliability of the tape heaters. The heaters arced and burned holes through the wall of the cold-steel drum. The arcing and resulting stray voltage found its way back to the computer and ruined the digital I/O card. The profile for this test resulted in a flat line, hence it is not shown. This was the last test that was performed using the tape heaters.

2.4.1.6 Test 6. For Test 6, band heaters were used. The 5-gal drum was filled with sand and buried under 610 mm (2 ft) of sand. Then, using the ISV ramp, the drum was cooked at 500°C for 24 hours. The profile for this test, which resulted in a flat line, is not shown.

Although the band heaters were only rated to 450°C, they worked at 500°C with no problems. Being buried in 610 mm (2 ft) of sand and covered with five layers of insulation only caused some discoloration to the band heaters. A problem was encountered when the setup became hot enough to melt the wiring to the heaters. Stainless-steel rods had to be used for wiring under the insulation. Because of the delay in receiving replacement heaters, some were used several times with no damage. However, when the sample reacted, it caused extreme temperatures both inside and outside of the drum.

2.4.1.7 Test 7. The sample material (87% base mix and 13% oil) reacted after heating for 358 minutes. This test was run unconfined, allowing video footage of the fixture. The video indicates that fire was venting from the vent pipe into the residue bucket causing the material to flame for a few minutes. The insulation was undamaged, but the aluminum-foil backing melted and fell to the ground, creating what looked like sparks on the videotape. Upon inspection, the sample material cooked the oil that was collected in the residue bucket.

The drum had completely disintegrated below the original fill line. Nothing was left but a rust-like material (probably ferric oxide). Some of the base mix seeped onto the dirt and some soaked into the insulation. The material on the ground was white and the material in the insulation was a

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b. "g" is defined as acceleration from gravity at the surface of the earth at 32 ft/second.[2]

greenish white. These residues were not further characterized. If not for the heaters and the chemical-soaked insulation, the drum would not have maintained its shape. In addition, the sidewalls were badly damaged and the bottom was completely gone. It was possible to poke a chisel through the rust into the bottom insulation (see Figures A-48 and A-49 of Appendix A). The bottom two heaters were damaged and no longer worked. All wiring and connectors were melted. As a result, the unit was bagged and stored for future reference.

The temperature profile (Figure B-8 of Appendix B) indicated that the test reached the set point of 500°C and remained there for about 1 hour before reacting. The inside wall (i.e., radius) temperature lagged the outside temperature. The inside wall temperature profile shows deterioration of the system (probably the sidewalls) at about 240 minutes. The outside wall temperature profile supports this supposition. The center of the material started to melt at about 280 minutes. There is no indication of a thermal runaway. At 358 minutes, the reaction (although not violent) occurred. Depending on whether mid-radius or center thermocouple readings were used, the temperature at which the reaction occurred was between 445 and 459°C, respectively. At this time, the thermocouples quit working and no other readings were acquired.

2.4.1.8 Test 8. The sample material (87% base mix and 6.5% oil with 6.5% CCl_4) started to melt at 323°C and 252 minutes into the test. The sample material reacted after heating for 318 minutes. Like Test 7, this test was run unconfined. The video indicates that smoke was venting from the vent pipe. The material smoked for several minutes and the vent gas temperature reached a high of 444°C. The insulation was undamaged, but there was some discoloration on the insulation at the bottom seam. Upon inspection, most of the material remained in the drum. The level of the material was about 63.5 mm (2.5 in.) lower than the original fill, however, this could be because of the physical change from a powder-pour density to a solid block of material formed from the molten salts.

The controller thermocouple failed at 512 minutes and then underwent a runaway reaction that reached 731°C. There were two benefits from this failure. First, we know that the heaters reached 731°C without destruction, and second, the material did not explode up to 690°C. At 603 minutes, the thermocouple resumed control of the heating cycle and lowered the temperature to 500°C, where it remained constant until the test was terminated.

The material that remained in the drum was a white solid with a tint of yellow. The level of the material was above the tips of the thermocouples.

Except for the runaway of the controller temperature to 731°C beginning at 512 minutes and regaining control at 800 minutes, the temperature profile indicated that the test reached the set point of 500°C and remained there for about 3,200 minutes. Figure B-10 of Appendix B is an expanded view of the test.

Air samples were taken until the concentration of the gas saturated the analyzer. Approximately 3 hours and 38 minutes into the test, the NO was 4,400 ppm, NO_2 was 1,160 ppm, CO was 1,000 ppm, and CO_2 was 30,000 ppm. Air sampling has not worked well because of the high concentration of gases in the air stream. A dilution of 100:1 (compressed air to gas) is as high as the system will handle.

2.4.1.9 Test 10. For this test, a mass of 35% cellulose (rags and Kimwipes) was treated with a saturated solution of base mix to give a 65-wt% ratio. The treated cellulose was damp at the beginning of the run. The interior of the drum was heated to 93°C at 72 minutes and soaked at this temperature for another 2.5 hours to evaporate the water. The vent temperature started to rise at 147 minutes into the test, indicating a reaction was starting somewhere in the drum. At 456 minutes, a reaction occurred with a peak temperature of 531°C. The profile indicates that the reaction was led from the inside of the drum

and not by a runaway heating system. Many spikes are in the temperature profile that would normally indicate electrical noise, but since the profiles are smooth after the reaction at 456 minutes, burning in the drum may have caused the temperature spikes (see Figure B-11 of Appendix B).

The 5-gal stainless steel drum was undamaged, except that the lid and bottom of the drum were bulged about 9.5 mm (3/8 in.) This would indicate that there was a rapid production of gases greater than could be released through the 38 mm (1.5-in.) vent. The heaters were undamaged and reused later in the series. A small amount of carbonized rags remained in the bottom of the drum and the inside wall of the drum was coated with a thin layer of white material. A violent reaction was not indicated by the video, the visual inspection of the test site, or the accelerometer data.

2.4.1.10 Test 11. For this test, 65% base mix was added to the top of a mixture of 35% dry rags and Kimwipes in a 5-gal stainless steel drum. The material reacted at 183 minutes into the test when the controller temperature was at 338°C. The sample was heating in a normal manner with no vent gas being produced, as indicated by the temperature profile (see Figures B-12 and B-13 of Appendix B). After the reaction, the temperature controller resumed the controlled heating at 500°C, but no further reaction occurred.

The test resulted in two temperature peaks (the first higher than the second) about 2 minutes apart. The center thermocouple reached its high of 903°C during the second peak. The test reaction escaped control at about 183 minutes into the test. The highest temperature reached was 1,103°C, recorded by both the radius and mid-radius thermocouples. The drum walls reached a temperature of 689°C during the second peak. The vent temperature peaked at about 700°C during the first peak. The interior temperatures of the drum started to react almost 30 seconds before the wall temperature began to rise. This would indicate a runaway reaction rather than a runaway heater.

The interior of the drum is shown in Appendix A (see Figure A-63). The material in the bottom of the drum is a white, hard material. Both the lid and bottom of the container were bulged out about 9.5 mm (3/8 in.). There was no apparent damage to the wall of the drum. The bulging indicates that the reaction generated gas faster than the gas could be vented through the 25-mm (1-in.) vent pipe.

Not the physical condition of the drum, the visual inspection of the test site, or the video footage indicated that an explosion had taken place. The accelerometer produced no viable data.

2.4.1.11 Test 14. This test consisted of 80% base mix placed on top of 20% carbonized rags and Kimwipes. The sample material reacted at about 230 minutes into the test. The controller temperature was 373°C and the interior of the drum was 273°C. About 2 minutes after the reaction began, the vent gas, controller temperature, and center temperatures reached 726, 957, and 1,165°C, respectively. Heating the material prior to the reaction was very even, although the interior temperatures trailed the outside wall temperatures. The heating rate was checked and calculated to be 101°C per hour over a 2.5-hour period (see Figures B-21 and B-22 of Appendix B).

The lid was not warped, but a 25-mm (1-in.) square hole was blown through the side of the container. The hole was located between the bottom and middle heater. The stainless steel flap from the hole remained attached along the left side, creating a flap standing at a 90 degree angle to the drum wall. The edges of the hole and the edges of the flap were fire polished, which indicate a hot flame within the container. The controller thermocouple, located just above the hole, was burned with only about 102 mm (4 in.) remaining.

Sand entered the container, either immediately after the rupture or during removal of the drum, which spoiled the chance to obtain a clear photograph of the sample material remaining in the drum. It

appeared that some solid, white material remained on the bottom of the container. It could not be determined whether any of the carbonized cellulose remained under the solid white material.

2.4.1.12 Test 15. This test was run on straight base mix in a cold-steel 5-gal bucket. The test was performed with no confinement. No accelerometer was used on this test. Since this test cooked for 4 days, the complete profile is about 9 MB and is too large to include in this text. Shown in Figure B-23 of Appendix B are the center, vent, and controller-temperature profiles, which are representative of the remaining profiles.

The center temperature (and all other locations), which reached 521°C, indicates a temperature rise starting at about 1,764 minutes. The temperature then dropped to 425°C, indicating that the thermocouples were no longer in the sample material. The radius thermocouple also failed at 1,764 minutes. At 1,764 minutes, the reaction resulted in a transfer of the liquefied material from the 5-gal bucket into the waste drum (located under the vent pipe) and into the atmosphere. The vent temperature reached approximately 225°C at this time.

When the test ended, the exterior of the drum appeared to be in good shape, with no holes or damage. However, the interior of the drum and lid were badly corroded with large flakes of rust peeling off. There is no evidence that a violent event took place during this test.

2.4.1.13 Test 16. For this test, 80% base mix was placed on top of 20% graphite and heated under confinement in a 5-gal stainless steel drum. Test 16 reacted in 236 minutes with a controller temperature of approximately 400°C (see Figures B-24 and B-25 of Appendix B).

The reaction melted the upper half of the bucket, leaving the heaters and insulation to maintain the drum shape seen in Figures A-74 through A-79 of Appendix A. The drum lid was totally melted and let the accelerometer stand, plus several gallons of sand, fall into the drum. Large holes were melted through the sides of the drum. This indicates that a very large amount of heat was liberated by the reaction. A visual inspection of the site indicated that the accelerometer stand had fallen about 254 mm (10 in.) from the original setup. The surface (i.e., sand) of the test was leveled before testing and the test resulted in a dish-shaped indentation, indicating that some sand dropped into the bucket. However, there were no indications that an explosion happened prior to the destruction of the lid.

The time and temperature profiles indicate that the reaction started about 236 minutes into the test. The vent temperature started to rise at 236 minutes and finally reached a peak of 1,232°C. The expanded profile shows that the peak temperature for the radius, mid-radius, center, and vent thermocouples reached 632, 1,056, 1,123, and 1,089°C, respectively. Beyond 240.18 minutes, the temperature readouts were cycling between 1,500 and 100,000°C, indicating that the thermocouples were destroyed by the reaction. Thus, the remainder of the data file was deleted because the cycling makes interpretation of the data unreliable.

There was no evidence from the condition of the drum, video footage, or the physical condition of the site that a violent reaction took place. The reaction shorted the heater bands, causing a power failure back at the transformer fuses, and the accelerometer software did not save the file. It is doubtful that a reaction would have been seen or recorded by the accelerometer. The software that records the temperature data normally saves files when the power fails, however, the uninterrupted power supply backup will only carry the computers for a limited amount of time.

Although the combination did not explode, an intense exothermic reaction occurred, resulting in the oxidation of the steel drum and the fusing of the surrounding sand and soil.

2.4.1.14 Test 18. This test consisted of rags and Kimwipes being treated with a saturated aqueous solution of base mix to give a nominal 40% cellulose to 60% base mix when dried. This material was air dried on screens, which was different from Test 10. The air-drying worked very well. The test was performed unconfined. This test reacted after168 minutes at a controller temperature of 316°C. No accelerometer was used on this test. See Figures B-30 and B-31 of Appendix B for temperature profiles.

This test confirms that the rather low-temperature reaction that took place has been seen in other tests of this same material. There was no gradual cook-off of the material and the sample reacted rather violently with no warning. Because the data collection rate was slow, the sample probably reacted quickly enough to either eject or destroy the thermocouples before they could show a temperature rise. Assuming that the sample material touching the wall was close to the same temperature as the wall, we can assume that the reaction temperature was close to 316°C.

The lid bulged upward and was completely blown off the drum. The bottom of the drum was bulged outward. The heaters did not appear to be damaged even though they were slightly stretched from the expansion of the drum.

2.4.1.15 Test 19. This was a test of 80% base mix with 20% graphite. This test reacted after 393 minutes with a controller temperature of 500°C. The controller thermocouple had been at 500°C for 133 minutes when the event occurred. This test burned quite vigorously and underwent a mild explosion, destroying all of the thermocouples within 6 seconds from the beginning of the reaction. The radial temperature peaked at 904°C. The vent temperature slightly preceded the rest of the temperatures and peaked at approximately 610°C. It is doubtful that any of the other thermocouples registered a peak temperature before they were destroyed by the reaction. The reaction caused the heaters to short, causing a circuit breaker ahead of the transformers to blow. This power failure resulted in the loss of the accelerometer data. The temperature collection software shut itself down properly and the profiles can be seen in Figures B-32 and B-33 of Appendix B.

Video footage of the test indicates that sand was blown into the air as the test vented. However, the accelerometer stand did not appear to be disturbed. Also, it can be seen on the video footage that smoke was being vented through the sand a considerable distance from the test. The lid was not melted, although venting gases moved it slightly upward, but the drum was badly damaged. Sand and residue also melted into large globs of glass, which were encrusted onto the accelerometer stand and around the lid.

2.4.1.16 Test 20. This test consisted of 60% base mix placed on top of 40% rags and Kimwipes material. The first reaction of this test occurred at 174 minutes with the controller temperature at approximately 320°C. It can be seen from the temperature profile in Figure B-34 of Appendix B that there were two temperature peaks.

The temperature profile shows that the radius thermocouple had a maximum temperature peak of 1,023°C during the first peak and approximately 960°C for the second peak. The top heater controller was the first to show a temperature rise, indicating that the reaction probably started in the upper part of the drum, most likely at the base mix and cellulose interface. Next to show a temperature rise was the vent thermocouple, indicating that the reaction was producing hot gases. The vent thermocouple reached a maximum temperature of approximately 550°C during the second peak.

Visual inspection of the drum showed no damage except for some discoloration. There was no evidence of a reaction at the surface, either from video footage or visual observation. There were some charred rags left in the drum after it had cooled, but the base mix was consumed by the reaction.

There was no evidence from the condition of the drum, video footage, physical observation of the site, or accelerometer data that a violent reaction took place. See Figure B-35 of Appendix B for the complete temperature profile.

2.4.1.17 Test 22. For this test, rags and Kimwipes cellulose was treated with a saturated aqueous solution of base mix to give a nominal 65 to 35-wt% nitrate and cellulose mixture after drying. The mixture was then placed in a 5-gal stainless steel drum and buried in sand to the lip of the drum. Next, the drum was heated to 150°C and the material was ignited with a resistance hot wire. See Figures B-39 and B-40 of Appendix B for the temperature profiles.

After 22 hours of heating, the internal temperatures of the sample were still not up to the intended temperature of 150°C. The temperatures of the radial, mid-radial, and center thermocouples were approximately 130, 85, and 76°C, respectively. An onsite decision was made to go ahead and do the test.

After ignition with the hot wire, the drum exploded instantaneously. The only thermocouple to register a temperature rise was the vent thermocouple, which peaked at approximately 100°C before the thermocouple line was broken. Obviously, this reaction occurred very rapidly, too rapidly for the thermocouples to register a temperature rise. However, since the lid was only bulged outward and blown off, and the drum was only bulged, this reaction was a deflagration and did not proceed to detonation.

The accelerometer data indicated that the initial shock was 71 "g". With the two camera views and the graduated 2 × 4-in. post, it was estimated that the accelerometer and stand were thrown approximately 9 m (30 ft) into the air. Somewhere during the trajectory, the cable to the accelerometer broke and the profile after the initial 71 "g" was reflecting the failure of the cable rather than the landing of the unit (see Figures B-41 and B-42 of Appendix B).

Most of the cellulose was consumed in the test. Some white residue was left in the bottom of the drum and some reacted (ash) and unreacted rags near the drum. The lid and drum were warped in the same manner as had been seen in previous tests.

2.5 Fifty-Five-gal Drum Cook-Off Tests

A "cook-off" test is the heating of a material, or combination of materials, to an elevated temperature to determine the possibility of runaway reactions. Variables may include the ratio of materials, target temperature, rate of heating, hold time at temperature, and amount of material, which affects the surface-to-volume ratio, and hence the ability to dissipate any heat generated. This is the reason for performing 55-gal tests after the 5-gal tests. This series of four tests, like most cook-offs, provided detailed data on any reactions that might occur under elevated thermal conditions. These tests consisted of 55-gal stainless steel drums, filled to 75% capacity of sample material, equipped with band heaters (with controller thermocouples), and heated. Each sample was tested individually in a pit surrounded by sand. The drum was covered with 2 ft of sand to furnish confinement similar to actual SDA conditions, except for Test 23, which was buried under 3 m (10 ft) of dirt and sand. The intended procedure was to heat the samples to 500°C, then hold at this temperature for approximately 10 days or until a reaction at an earlier time. An ISV heating rate of 100°C per hour was the target, but the mass of material in the drum lagged the rapid rate produced by the outside walls.

Data were collected in the form of (1) internal temperatures of the sample material during the entire test, (2) video footage used to observe any physical changes at the surface, and (3) accelerometer data should an explosion occur. During the soak phase at 500°C, the test site was visited once per day to check the equipment and rewind the tapes.

Two types of heaters were used for the tests, depending on availability. The heaters were either custom-built, three-phase ceramic band heaters purchased from Industrial Heater Corporation, or Watlow Model 125H223A1A cable heaters purchased from Cybernetics. Test 17 was the only successful test run using the ceramic band heaters. Tests 21 and 23 used cable heaters. Test 13 was started three times using the ceramic band heaters, but was only successfully run using cable heaters. Six cable heaters were used for each test and wrapped in a nonoverlapping spiral over the whole length of the drum. Each heater was 3.2 mm (1/8 in.) in diameter × 5.7 m (223 in.) long.

For the tests using ceramic band heaters, one control thermocouple per heater was used. For the tests using cable heaters, two thermocouples were used to control the rate of heating. One heater was placed on the lower third of the drum with a second located toward the top of the middle third, with both thermocouples placed under different heaters. Three thermocouples, 6.4 mm (0.25 in.) in diameter × 610 mm (24 in.) long, were placed through the container lid and extended 559 mm (22 in.) into the drum. Longer thermocouples would have been used, but with an 8-week delivery, the testing was performed around availability. The interior thermocouples were placed in the center (axial), mid-radius, and radius (close to the inside wall).

A 38-mm (1.5-in.) vent pipe was installed into the lid of each test. Using a system of elbows and nipples, any material being ejected from the experiment (in the form of vapor or liquid) would be contained in a 55-gal waste drum. The waste drum was designed to protect the environment and to allow for collection, sampling, and measuring of sample materials vented during the test. One thermocouple was located in the vent pipe to sample the temperature of the vent gases.

Each test was wrapped with three layers of 8-lb, high-density fiberglass insulation and two layers of foil-backed glass insulation of the same density. Glass tape was used to keep the insulation in place until the final layer was wrapped and taped with duct tape.

An accelerometer was mounted on a 762-mm (30-in.) stand placed on the lid of each test. Four Mode-3010 accelerometers were purchased from PCB Industries and were calibrated before leaving the factory. The accelerometer was connected to a second Pentium computer containing a similar Dynares interface board. The data were collected using *Workbench for Windows* software (IOtech/Strawberry Tree 1996) at 100 Hz. Faster would have been better, but the largest file was 84 MB and 2.34-million lines long. Files of this size and larger were hard to handle.

Test 23 was set up the same as previous tests except that it was buried deeper. The top of the drum was buried under 2 m (7 ft) of dirt (to the ground-surface level), with 3 ft of sand used as the final cover (aboveground), to complete a 3-m (10-ft) fill above the drum lid. The flat-topped sand cover was approximately 2.4 m (8 ft) across the top and 3.7 to 4.3 m (12 to 14 ft) across the bottom. The bottom diameter of the sand was larger than the top diameter of the hole to prevent blow-by at the soil-surface interface.

In addition to attaching the accelerometer to a 762-mm (30-in.) stand placed on the drum lid before burial, a second accelerometer was mounted on a 3.2-mm (1/8-in.) thick by 356-mm (14-in.) square aluminum plate placed on the surface of the sand above the drum lid. Two pressure transducers were placed above the drum lid. The lower transducer was 305 mm (12 in.) above the surface of the sand with the second 914 mm (36 in.) above the sand. The lower transducer was a 50-psig Model 102A07 and the upper an 8.2-psig Model 106B. Both were purchased from PCB Piezotronics. Calibration of the 102A07 was 93.7 mV/psi and the 106B was 320.5 mV/psi.

Two cameras, one located about 15 m (50 ft) away and the other about 91 m (300 ft) away, were used to record the explosion. A 2.4 m (8 ft) long 2 x 4-in. post, with 305-mm (1-ft) graduations marked on it, was placed 1.2 m (4 ft) up the transducer stand for scale. Two microphones were also used.

2.5.1 Sample Mixtures

2.5.1.1 Test 9. An air (empty drum) pressure test was conducted. This test contained no mixture and because the drum was empty when the pressure was applied, no accelerometer data were obtained for the test.

2.5.1.2 Test 13. A mixture of 90% base mix and 10% Texaco Regal R&O machine oil was tested. The procedure was designed to completely mix the base mix and oil in totes just before loading the drum. Consequently, the procedure decreased the settling (or degradation) of the oil mixture.

2.5.1.3 Test 17. A saturated aqueous solution of the nitrate base mix was added to a 50% by weight mixture of rags and Kimwipes in a quantity sufficient to give a nominal 63 to 37-wt% nitrate and cellulose mixture when the water was evaporated.

2.5.1.4 Test 21. A quantity of the nitrate-base mix, sufficient to give a nominal 86 to 14-wt% nitrate and carbon ratio, was placed on top of unground, carbonized-cellulose material prepared by the pyrolysis of a 50% by weight mixture of rags and Kimwipes.

2.5.1.5 Test 23. A quantity of the nitrate-base mixture, sufficient to give a nominal 80 to 20-wt% nitrate and carbon ratio, was placed on top of unground, carbonized-cellulose material prepared by the pyrolysis of a 50% by weight mixture of rags and Kimwipes.

See Appendix A for pictures of the tests discussed above. The results are listed in Table 4.

2.5.2 Results

2.5.2.1 Test 9. The purpose of Test 9 was to determine the burst pressure of a 55-gal drum. The drum was attached to a bottle of compressed air with a regulator to vary the pressure in the drum throughout the test. Also, the drum had a 500-psi Setra pressure transducer hooked up to the incoming airline near the drum. An accelerometer was attached to the lid of the drum to measure the acceleration of the lid in case the drum (catastrophically) failed. The numbers on the bottom of the drum were:

- UN1A2/Y15/100/99
- UN1A2/Y415/5/99
- 12/9/12/208L.

The drum held 38 psig of air until the lid seal failed at the retaining-ring bolt. The drum did not fail in a spectacular fashion, it just sprung a leak. However, both the lid and the bottom of the drum bulged outward. No accelerometer data were obtained for this test.

2.5.2.2 Test 13. This test was set up and buried four times before a successful test was performed. About 5 hours into the first test, a lightning strike forced the shutdown of the test. The strike took out the electronic relays, two Dynares boards, a video-cassette recorder, and one computer. The heaters did not seem to be damaged. Energetic Materials Research and Testing Center personnel (listed in Section 5) suggested that the lightning strike damaged the transformers, however, the transformers tested

satisfactorily under a light load. The power company suggested that the heaters were faulty or connected wrong. The last three 55-gal tests were moved to a different test location and performed with no problems, using cable heaters. The test was restarted after replacing the damaged equipment, but the electronic relays repeatedly malfunctioned after about four hours.

The test was set up using three-phase ceramic band heaters. The heaters were changed out two more times before a decision was made to use single-phase cable heaters. The cable-heater setup had some trouble, but finally the test was successfully run. Total time to perform Test 13 was 4 weeks.

Test 13 was allowed to run for 7 days at 500°C and no violent event occurred during that time period (see Figures B-19 and B-20 of Appendix B). A small thermal event occurred when the vent temperature ramped to 416°C at 2,125 minutes. This ramp may indicate volatilizing the oil because the waste collection drum was about 25% full of oil and other materials when the test ended. The internal thermocouples showed that no thermal event occurred. This was very similar to the thermal tests performed on the 90% base mix mixture and 10% oil mixture at the 5-gal size. Those tests resulted in no explosions, however, they did catch fire and severely oxidize the drum in Test 7.

After the mixture was cooked for 7 days, and because previous tests at the 5-gal level showed no reactions, it was decided to shut the test down to allow the cabling, relay box, and video equipment to be moved for the rest of the series. The cooldown was very slow and continued for another week.

After the test had cooled, it was dug up and samples were taken for possible analysis. The remainder of the salt mixture left in the 55-gal drum was destroyed, using 45 kg (100 lb) of ammonium nitrate fuel oil (ANFO) and a C-4 booster. The residue from the test was destroyed by explosion, because after heating it could have become much more sensitive to accidental explosion. The destruction took place on the surface of the test pad. Without the proper instrumentation, it cannot be determined whether a partial detonation occurred during the intentional destruction. However, a partial or dying detonation would not be unusual, because other nitrate salts such as NH_4NO_3 would detonate without any fuels being added, assuming the salt was of sufficient quantity and was shocked hard enough. The shock did not leave a hole in the ground indicating that any resulting detonation died before reaching the bottom of the drum. Observers suggest a partial detonation because of the loudness of the sound. Without instrumentation and a calibration test, this method proves to be unreliable for determining a partial detonation.

2.5.2.3 Test 17. This test reacted rather violently after 124 minutes with the control temperature at 209°C (see Figures B-26 and B-27 of Appendix B). There was no detonation, only an explosion that removed the lid from the barrel and placed it beside the hole. With the lid blown off, sand caved in and filled the barrel. Some of the rags were not consumed in the reaction and were blown to the surface or left in the drum. The barrel was dumped and the unreacted material sorted out for disposal. Chemical residues left from the reaction were mixed with sand and were not recoverable. Neither pictures nor samples of any residue left in the drum were possible because the barrel was filled with sand.

Although the top was removed by the eruption, the ring remained clamped on the drum. Both the top lid and the bottom of the drum were bulged outward by pressure from the explosion before the lid pulled loose from the clamp. This would indicate that the reaction was a deflagration and did not proceed to detonation. (See the discussion of explosion, deflagration, and detonation in Section 3.8.) No sample material was vented into the waste drum. The explosion damaged the top heater, but according to the time and temperature profile, the bottom heater seemed to be working. This was the only successful test done with the ceramic band heaters.

Extra care was taken when examining the barrel to determine whether an electric heater had shorted to the stainless steel drum, thus causing the reaction. There are no markings on the drum to indicate an electrical short and the computer-interface board was undamaged. If the interface board were damaged, it would indicate the presence of a high-voltage short between the band heater and the drum.

The expanded view of the profile explains nothing about the reaction except that the thermocouple wires were pulled loose and were being moved about by the reaction. There was no temperature rise in any of the thermocouples before the reaction, indicating that the reaction occurred very quickly and ejected the thermocouples before they could register a temperature rise.

The lid moved with an acceleration of 74 "g" (disregard the secondary peaks in the graph [Appendix B] caused by the landing of the accelerometer and mounting stand). No attempt should be made to match the timing between the temperature and accelerometer data. Separate computers collected the data and starting them at exactly the same time (within 0.1 second) by two operators was impossible. On the profiles, time was measured at 124.097 minutes for the accelerometer and compared to time measured at 124.0 minutes for the temperature. One cannot determine exactly where on the temperature profile the lid came off (see Figures B-28 and B-29 of Appendix B for the acceleration profile).

2.5.2.4 Test 21. This test exploded 198 minutes into the test, at a controller temperature of 366°C. The explosion removed and threw the lid about 3 m (10 ft) to the side. Some of the carbonized cellulose was blown out of the drum and onto the surface of the bank. Since the drum accumulated about 304 mm (1 ft) of sand, the amount of sample material remaining could not be determined.

The reaction in this test was similar to the reaction in Test 17, except that it was a little more vigorous, more sand was moved, and the lid was thrown farther. Again, the internal thermocouples showed no temperature spike before the reaction. However, the top controller thermocouple did show a temperature increase preceding the reaction. This might indicate that the reaction occurred near the top of the barrel, most likely at the base mix and carbonized cellulose interface. Unfortunately, the leads for the vent thermocouple were unintentionally reversed during this test. However, it can be seen from the temperature profile that the vent thermocouple temperature went negative at the same time as the temperature spike from the top controller thermocouple. This temperature drop indicates a temperature increase for a thermocouple with reversed leads. In addition, hot gases were being vented at this time. See Figures B-36 and B-37 of Appendix B for temperature profiles.

The accelerometer gave an acceleration of 79 "g," which was slightly higher than Test 17. For this test, the accelerometer data were recorded at 200 Hz, resulting in a data file with 2.4 million lines of data. Since it was a flat line until the reaction, the unneeded data were discarded. Again, the secondary peaks should be ignored (see Figure B-38 of Appendix B).

The actual physical condition of both the 55-gal drum and the waste drum indicated, more than any other factor, that this test was more violent than Test 17. The test drum was pushed to the surface with only about 304 mm (1 ft) of the drum being buried in the crater. Both the top and the bottom of the drum were bowed outward, but the 55-gal drum-locking ring remained in place. The waste drum was also damaged in this test. The top of the waste drum was bulged outward and blown off and the bottom was bulged outward as well. This would indicate that reaction gases were vented into the waste barrel quite vigorously through the vent pipe.

2.5.2.5 Test 23. This test reacted violently after 387 minutes with a controller temperature of 500°C. The controller temperature had been at 500°C for approximately 1 hour at the time of the event. The 914-mm (3-ft) sand pile was cratered off-center down to the level of the ground, with one side of

the sand pile being slightly higher than before the event. Sand, hot ash, and carbonized cellulose was vented about 9 m (30 ft) from the side of the hole.

The reaction of this test was similar to tests 17 and 21 except that it reacted at a much higher temperature. There were no sudden temperature spikes in the internal thermocouples even though they were at higher temperatures than in tests 17 and 21. The radial thermocouple was at 460°C, but the center had only reached 87°C. There was an error with the bottom thermocouple causing it to read at a significantly higher temperature than it should have. Either the calibrated thermocouple was producing erroneous readings, or the heater was uncontrolled. The temperature spike of the controller thermocouples, at approximately 100 minutes, resulted from switching the control of all heaters to the top controller thermocouple. There was no significant increase in the vent temperature (see Figure B-43 of Appendix B).

The lower accelerometer on the steel tripod registered 26.5 "g" acceleration, while the upper accelerometer on top of the sand did not give any viable data. Again, any secondary peaks in the accelerometer data should be ignored. The accelerometer stand was found approximately 610 mm (2 ft) below the level of the ground. The accelerometer was no longer attached to the stand and the accelerometer cable had broken. The accelerometer was never recovered. See Figure B-44 of Appendix B for acceleration and pressure data.

The bottom pressure transducer, 305 mm (12 in.) above the sand, measured a peak pressure of 2.4 psig. There were some peaks later on, but most likely these were only sand impacting the transducer. The upper transducer, 914 mm (36 in.) above the sand, showed no reaction to the event.

Again, a major difference between Test 23 and the other tests was the damage done to and movement achieved by the drum. The resulting explosion raised the drum 1.2 m (4 ft) upward and bulged both the drum and lid several inches outward. The drum was turned partially sideways and crushed to an oval shape with one flat side. The drum became filled with sand and some unreacted carbonized cellulose. The insulation and parts of the cable heaters remained buried below the drum. The lid was found just above the drum and to the side, with the thermocouples bent against the bottom of the lid. The locking ring was found (severely deformed) approximately 610 mm (2 ft) from the surface. The severe deformation of the locking ring may have been caused by the backhoe while digging up the test site. A waste drum was not used during this test. What caused the large movement of the drum remains unresolved. Since the drum moved and the insulation remained in place, this suggests that the pressure wave caused by the evacuating gases resulted in an underpressure behind the drum, which pulled the drum along. This, however, remains open to conjecture. In addition, the damage done to the drum suggests this was quite a violent event.

2.6 Fifty-Five-gal Drum Containment and Mitigation Tests

2.6.1 Introduction

The following tests were performed to determine, under "realistic" conditions, the effectiveness of mitigative measures (i.e., a 2-m [7-ft] depth-of-burial plus an additional 914-mm [3-ft] dirt cap). The burial of the drum in Test 23, 2 m (7 ft) beneath the surface in a well-like hole, created a chimney effect. The blast effects were directed straight upward, much like in a mortar or gun barrel. For the subsequent tests, the hole was excavated with a much greater taper so as to give a larger area and mass of uniformly compacted material above the drum, with a total 3-m (10-ft) overburden.

2.6.2 Test Procedure

2.6.2.1 Test 24. A drum full of dry rags and Kimwipes was subjected to pyrolysis conditions by heating at approximately 400°C. The drum was cooled to ambient temperature and weighed. The calculated quantity of nitrate mixture needed to give a stoichiometric mixture of 85% nitrates and 15% carbon was then placed on top of the carbonized material, and the drum was heated to 500°C beneath 3 m (10 ft) of overburden. A video camera was set up to detect any heaving of the surface above the drum. Accelerometer and peak overpressure data were collected. Following the test, the surrounding area was inspected for material ejected from the drum.

2.6.2.2 Test 25. A drum full of air-dried, nitrate-soaked rags (65% nitrates and 35% rags) was heated to 500°C beneath 3 m (10 ft) of overburden. A video camera was appropriately placed to detect any heaving of the surface above the drum, and accelerometer and peak overpressure data were also obtained. Following the test, the surrounding area was inspected for material ejected from the drum.

2.6.2.3 Test 26. The stoichiometric quantity of nitrate mixture, to give an 85 wt% to 15% carbon ratio, was added on top of graphite fines. The drum was then heated to 500°C beneath 3 m (10 ft) of overburden. A video camera was placed to detect any heaving of the surface above the drum, and accelerometer and peak overpressure data were also obtained. Following the test, the surrounding area was inspected for material ejected from the drum.

2.6.2.4 Test 27. This test was similar to Test 1, but included an overnight hold (i.e., soak) at 250°C and a tracer (e.g., terbium oxide) to determine whether any of the sample material had vented during the pressure explosion. Soil samples were taken before and after the test, and have been received at the INEEL. The samples were to be analyzed in conjunction with ISV samples during the ISV cold test conducted during the summer of 2001. Only four mitigation tests were performed as Phase 4 of the project.

2.6.3 Experimental

The following four tests were buried 2 m (7 ft) below grade, with an additional 0.9-m (3-ft) cap. The only previous test buried 3 m (10 ft) deep was Test 23, which contained more material and breached the surface.

Cold-steel 55-gal drums were used for this test series. These drums were purchased from a commercial packaging supplier. The drums had removable tops, locking rings, and were stamped as approved DOT shipping containers. After the drums were filled with the sample material, the lid and locking ring were installed. A calibrated torque wrench tightened the ring to 75 lb/ft.

Data were collected in the form of (1) internal temperatures of the sample material during the entire test, (2) video footage to observe any physical changes at the surface, and (3) accelerometer data to record any movement of the drum lid should an explosion occur. During the soak phase at 250°C, the test site was visited several times to check the equipment and rewind the tapes.

Six cable heaters were used for each of the mitigation tests and wrapped in a nonoverlapping spiral over the whole length of the drum. Each heater was 3.2-mm (1/8-in.) in diameter × 5.7 m (223 in.) long.

Two thermocouples were used to control the rate of heating. One was placed on the lower third of the drum with a second located toward the top of the middle third, with both thermocouples located under different heaters. Three thermocouples, 6.4-mm (0.25-in.) diameter by 914 mm (36 in.) long,

were placed through the container lid and extended 559 mm (22 in.) into the drum. The interior thermocouples were placed in the center (axial), mid-radius, and radius (close to the inside wall).

Each test in the mitigation series (i.e., tests 24 through 27) was wrapped with three layers of 8-lb, high-density fiberglass insulation and two layers of foil-backed glass insulation of the same density. Glass tape was used to keep the insulation in place until the final layer was wrapped and taped with duct tape.

The temperature data were collected in the same manner as the 55-gal cook-off tests.

An accelerometer was mounted on a 762-mm (30-in.) stand placed on the lid of each test. Four 50G Model 353B33 accelerometers, each having a range of 0 to 50 "g", were purchased from PCB Industries and were calibrated before leaving the factory. For Test 4, a 250G Model 353B01, having a range of 0 to 250 "g", was purchased. The accelerometer and pressure transducers were connected to a second Pentium computer containing a similar Dynares interface board. The data were collected using *Workbench for Windows* software at 200 Hz. A faster recording speed would have been better, but at this speed, the largest file had 2.2 million lines of data. Files of this size and larger were hard to handle.

Two pressure transducers were placed above the drum lid. The lower transducer was 305 mm (12 in.) above the surface of the sand, with the second being 914 mm (36 in.) above the sand. The lower transducer was a 50-psig Model 102A07, and the upper an 8.2-psig Model 106B. Both were purchased from PCB Piezotronics. Calibration of the 102A07 was 93.7 mV/psi, and the 106B was 320.5-mV/psi.

As with the 55-gal tests, two video cameras were used to record the explosion. One was located about 15 m (50 ft) away from the testing area and the other (i.e., the surveillance camera) about 91 m (300 ft) away. A 2.4-m (8-ft) long 2 x 4-in. post, with 305-mm (1-ft) graduations marked on it, was used to measure the height of any eruptions. A stake was driven into the soil surface (i.e., cap) directly above the drum for post-test reference.

All four tests were buried under 2 m (7 ft) of compacted dirt to a grade level with the surrounding soil surface. The dirt was compacted using the backhoe bucket. A 914-mm (3-ft) non-compacted cap of dirt was added to cover the entire hole and the surrounding soil surface. A 7.3-m- (24-ft) diameter cap proved sufficient to prevent venting of the test. A layer of sand was placed around and over the barrel and accelerometer to protect the instrumentation from the stress of the soil compaction.

2.6.4 Sample Material

2.6.4.1 Test 24. A 50% cotton rag and 50% Kimwipe mixture was placed in a drum and heated at 400°C for 6 hours (until carbonized), then cooled and weighed. The weight ratio of initial rags to obtained carbon was about 3 to 1. For this test, 30,888 g (68 lb) of rags and Kimwipes resulted in 9,271g (20.4 lb) of carbon. The carbon was placed in the drum and topped with 58,314 g (129 lb) of base mix, which contained 52,481 g (116 lb) of nitrates.

2.6.4.2 Test 25. For Test 25, 10,035 g (22 lb) of rags and Kimwipes were soaked with a nitrate solution and dried. The solution required 20,631 g (45.5 lb) of base mix containing 18,568 g (41 lb) of nitrates.

2.6.4.3 Test 26. Test 26 placed 175,951 g (388 lb) of base mix (containing 158,355 g [349 lb] of nitrates) on 27,945 g (62 lb) of commercial graphite that had been placed in the bottom of the drum.

2.6.4.4 Test 27. Test 27 used the same procedure as Test 24, except 30,897 g (68 lb) of rags and Kimwipes resulted in 9,485 g (21 lb) of carbonized rags and Kimwipes. On top of the carbon, 59,723 g (132 lb) of base mix (53,751 g [118 lb] of nitrates) was added. Terbium oxide (843 g [1.9 lb]) was layered throughout the carbon.

2.6.5 Results

2.6.5.1 Test 24-15% Carbonized Rags and 85% Nitrates. The experiment reacted 174 minutes into the test with the controller temperatures near 330°C. The reaction temperature profile indicates the reaction lasted for less than 0.15 seconds before the thermocouples stopped working. The radius thermocouple did not register a peak (see Figure B-45 of Appendix B). This could have been caused by the thermocouple and the reacting mass being some distance apart (i.e., the explosion could have taken place before the thermocouple could react).

The acceleration profile (see Figure B-47 of Appendix B) reached a peak of 103 "g". The expanded view has the profile with a somewhat flat top (see Figure B-46 of Appendix B), suggesting that the acceleration exceeded the maximum value that the accelerometer would measure (100 "g"). This suggests that the actual acceleration was somewhat higher and could be projected around 120 "g". The damage to the drum and the lid would suggest considerable thrust caused by the explosion.

The lid was blown off the barrel and bulged upward. An imprint of the accelerometer stand was clearly visible. The edges of the lid were bent down, the accelerometer stand was bent, the barrel was bent and ruptured, and the bottom was bulged. A nonbreaching crater was created 1.1 m (3.5 ft) below grade. Excess carbon remained in the crater and barrel.

There was no obvious breach of the soil cap. It was not clear whether any cracking of the cap surface occurred. The surface of the cap was semismoothed dirt and interpretation of any surface expansion was difficult. A brushed-sand surface was added to the cap in later tests to facilitate the determination of surface cracking.

2.6.5.2 Test 25-35% Rags and 65% Nitrates (Soaked and Dried). The nitrate-soaked and dried rags reacted after being heated for 125 minutes. The test exploded at 244°C outside wall temperature. The highest sample temperature recorded was 72°C. It is obvious that a hot spot ignited the drum to start the reaction, but the reaction did not start near the interior thermocouples. The temperature fluctuations seen in Figures B-48 and B-49 of Appendix B are noise caused by damage to the thermocouples and data lines.

The accelerometer data made a flat-topped profile at 115 "g". The profile would suggest some trouble with the data system. The pressure transducer, located 610 mm (2 ft) above the surface of the cap, recorded a peak reading of 68 psig (see Figures B-50 and B-51 of Appendix B). Both of the profiles indicate problems. The pressure peak was not believable, considering the soil surface was not disturbed. Replacement pressure gauges were installed.

The lid was blown off the barrel and bulged upward, though the lock ring remained on the drum. An imprint of the accelerometer stand, which was bent, was clearly visible in the drum surface. The bottom of the barrel was bulged, but the barrel remained intact. Most of the material was consumed in the reaction. A spider-web-crack pattern was observed about the stake in the brushed-sand surface of the cap.

2.6.5.3 Test 26-15% Graphite and 85% Nitrates. A soak period was used for the graphite test. The drum was heated to 200°C and soaked at this temperature for 21 hours before letting the 100°C per

hour heating rate start. The ramp started at 1,361 minutes (see Figures B-52 and B-53 of Appendix B). At 1,570 minutes, and with one of the controllers at 501°C, one of the controllers and the heaters connected to it stopped working.

The system was shut down to run as many system checks as could be run without approaching the test. It was found that only one controller and one heater were operational. The test was restarted and allowed to heat until a reaction occurred at 3,054 minutes at an outside wall temperature of 500°C. The interior of the drum reached approximately 402°C before the reaction occurred.

This test with graphite would be considered a thermal reaction rather than an explosion. The lid, lock ring, and drum melted down into a clump about 33% of their original size. A portion of the accelerometer stand also melted into the clump. The sand around the barrel was melted into a glass-like mass. A crater was created below grade, but the distance-to-crater is not known because the front wheel of the backhoe caved in the roof of the crater. Brushed-sand surface cracking was not observed.

Neither the accelerometer nor the pressure transducers reached peaks that could be reliably sorted from the noise. See Figure B-54 of Appendix B for the accelerometer and pressure transducer data.

2.6.5.4 Test 27-15% Carbonized Rags and 85% Nitrates (with Terbium Oxide Tracer).

Test 27 was also performed with an initial soak period. The controller temperature was raised to 250°C and the test was soaked at this temperature for approximately16 hours before the temperature ramp of 100°C per hour was started. An explosion occurred 41 minutes later, at 314°C. The interior temperature of the barrel varied from 210°C (mid-radius) to 240°C at the radius. The controller thermocouple recorded a peak temperature of approximately 1,100°C at the event, although the interior thermocouples only reached 345°C before the data lines were severed. See the temperature in Figure B-55 of Appendix B and the expanded profile in Figure B-56 of Appendix B.

A new, 250-g accelerometer was used for this test. The peak acceleration was recorded at 2,800 "g" and the peak pressure at 544 psig. Neither of these values is believable. Perhaps stray voltage from the broken band heaters through the AD converter affected the computer readings (see Figure B-57 of Appendix B).

The lid was blown off the barrel and rested offset about 25 cm (10 in.) over the barrel edge. The lid was bulged upward and an imprint of the bent accelerometer stand was visible. The bottom of the barrel was bulged outward, but the barrel remained intact. A crater was created just below grade, with excess carbon observed in the crater and barrel. White solids were observed in the insulation around the barrel and surrounding sand. A crack 54-cm (22-in.) long ran north to south about the stake in the brushed-sand surface of the cap.